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Bimodal Microstructure and Mechanical Properties of Cryomilled Nanocrystalline Al-7.5Mg

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ABSTRACT

The microstructure and mechanical properties consisting of tensile behavior and hardness of bulk nanocrystalline Al-7.5Mg alloy were investigated. Grain refinement was achieved by cryomilling of atomized Al-7.5Mg powders, and then nanocrystalline powders blended with 15% and 30% coarse-grained Al-7.5Mg powders were consolidated by hot isostatic pressing (HIP) followed by extrusion to produce bulk nanocrystalline Al-7.5Mg alloys. Bimodal structures, which enhance ductility and toughness of nanocrystalline metals, were produced that consisted of nanocrystalline grains and elongated coarse-grain bands. Examination of indentation revealed unusual deformation mechanisms and interactions between the coarse-grain bands and nanocrystalline regions. The ductile coarse-grain bands underwent extensive plastic deformation near indentation, while nanocrystalline regions exhibited limited deformation.

INTRODUCTION

The relations between microstructures and mechanical properties of bulk nanocrystalline metals have been investigated in recent years. Although several materials and microstructures have been developed high strength nanocrystalline metals, nanocrystalline metals still suffer from ductility and toughness reduction in contrast with increasing its strength. In one recent study, elastic-nearly perfectly plastic stress-strain behavior was observed and reported in Al-10Ti-2Cu alloy produced by mechanical alloying using cryomilling [1,2,3]. Such behavior is atypical for coarse-grain alloys of similar composition. Multi-scale structures, which were consisted of nanocrystalline grains and coarse-grain regions, enhanced ductility and toughness of bulk nanocrystalline alloys. In an effort to optimize toughened alloys based on multi-scale microstructures, more thorough understanding of microstructure and of structure-property relations in this structure was required. In the extended study, bimodal structures, which were comprised of nanocrystalline grains separated by coarse-grain regions, were produced to achieve ductile phase toughening in Al-7.5Mg. Thus in this work, the bimodal microstructure of bulk nanocrystalline Al-7.5Mg alloy was investigated using electron microscopy and optical microscopy. The stress-strain behaviors of bimodal Al-7.5Mg were estimated using uniaxial tensile test. In addition, Vickers hardness tests also were performed along the parallel and perpendicular to the extrusion direction to investigate the orientation dependent response of the bimodal microstructures and the interactions between coarse-grain and nanocrystalline regions.

EXPERIMENTAL DETAILS

Spray atomized Al-7.5%Mg in weight percent alloy powders were used to produce materials for this work. Nanocrystalline powder was produced using low-energy mechanical attrition at a cryogenic temperature (cryomilling) with a stainless steel vessel and milling balls with a diameter of 6.4 mm. The ball to charge ratio was 36:1, with stearic acid added at 0.25 percent of the powder weight to moderate the cold welding process. The cryomilling was operated at 180 rpm for eight hours, and maintained at a temperature of −190 °C using flowing liquid nitrogen into the vessel itself, submerging the powder and balls. Cryomilled nanocrystalline powders were combined with coarse-grained Al-7.5Mg powders of 0, 15 and 30 weight percent respectively and were blended to form bimodal samples. These samples were loaded in aluminum cans for vacuum degassing at 400 °C. The cans were sealed and consolidated in hot isostatic press (HIP) at 325 °C and 25 ksi. The consolidated billets were extruded using an extrusion ratio of 6.5:1 and proprietary rate and temperature parameters.

Tensile specimens were machined from the resulting extrusions along the extrusion direction with a gauge length of 13.5 mm and a gauge diameter of 3.5 mm. The tensile tests were performed on a universal testing machine at room temperature, using a nominal strain rate of 10^{-3} s^{-1}. The microstructure of the extrusions was examined using transmission electron microscopy (TEM) and optical microscopy. Vickers hardness measurements were performed on the polished and chemically etched surfaces along the extrusion and transverse directions at 10 gf load for the coarse-grain region and nanocrystalline region respectively, and 1 kgf load for the global region, which covered both coarse-grain and nanocrystalline region. The polished and chemically etched surfaces and indentations were observed using optical microscope and scanning electron microscopy (SEM; Cambridge 360). Thin foils for transmission electron microscopy (TEM) were prepared by tripod polishing to prepare an electron transparent wedge, which provided large thin areas for TEM observation. Samples were examined using a Philips EM420 TEM with EDS. In order to reveal both global and local structure, TEM images were recorded at low and high magnifications using Philips EM420 and Akashi 002B TEMs.

DISCUSSION

The bimodal microstructures comprised of nanocrystalline grains and coarse-grains were prepared by consolidation of powders and extrusion successfully. Bulk nanocrystalline Al-7.5Mg mixed with 15% and 30% coarse-grain had bimodal structures with nanocrystalline region and coarse-grain regions. However, the microstructure of Al-7.5Mg with 0% was uniform with a few percent of residual coarse grains. The global three-dimensional view of bimodal structure of the as-extruded Al-7.5Mg with 30% coarse-grain is shown in figure 1. Coarse-grain regions and nanocrystalline regions are evident in the figure. The bright and dark regions indicate coarse-grain and nanocrystalline regions, respectively, on the figure. Both coarse-grain and nanocrystalline regions extended along the extrusion direction while coarse-grain regions extended in highly elongated bands. The resulting bimodal structures were comprised of nanocrystalline grains separated by coarse-grain bands. The coarse-grain bands, which were dispersed uniformly and were similar in their shape and size, were about 240 μm long and 20 μm wide in Al-7.5Mg with 30% coarse-grain. The spacing, which occupied with nanocrystalline, between coarse-grain bands was about 25 μm. The length and width of coarse-grain bands of Al-

7.5Mg with 15% coarse-grain were shorter than those of Al-7.5Mg with 30% coarse-grain and the spacing between coarse-grain bands of Al-7.5Mg with 15% coarse-grain was bigger than that of Al-7.5Mg with 30% coarse-grain. The coarse-grain bands usually consisted of grains and sub-grains about 1 μm in contrast with nanocrystalline region, which had grains about 200 nm sizes. There were no intermetallic phases detected in both coarse-grain bands and nanocrystalline region. Figure 2 shows the microstructures of the coarse-grain bands and the sub-grains within Al-7.5Mg with 0%, 15% and 30% coarse-grain, respectively. The interfaces between nanocrystalline region and coarse-grain bands looked rather faceted. The ductile coarse-grain bands and nanocrystalline region had the same composition of Al-7.5Mg.

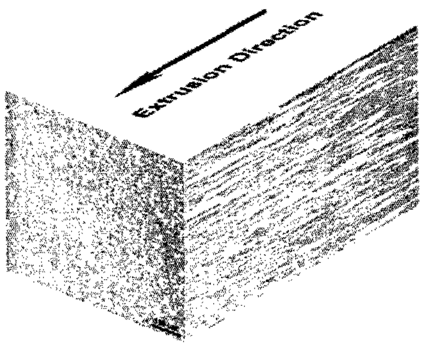

Figure 1. Three-dimensional view of bimodal microstructure of as-extruded Al-7.5Mg alloy with 30% coarse-grain content.

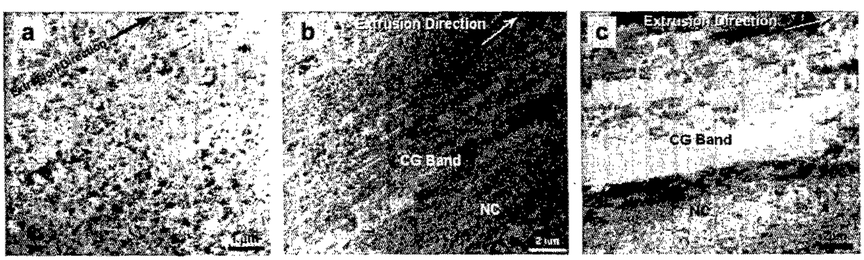

Figure 2. TEM micographs of as-extruded Al-7.5Mg alloy with 0% (a), 15% (b) and 30% (c) coarse-grain content, respectively.

The engineering stress-strain curves obtained from uniaxial tensile tests along the extrusion directions are displayed as a function of coarse-grain content in figure 3. The tensile stress-strain behavior of the Al-7.5Mg with 30% coarse-grain alloy exhibited elastic-nearly perfectly plastic stress-strain behavior in noticeably contrast to that of the Al-7.5Mg with 0% coarse-grain, which showed limited plastic deformation and work hardening. This feature is consistent with the

uniaxial tension stress-strain behavior reported for sintered nanocrystalline Al-Ti-Cu [1,2,3] and Al [5]. The yielding was followed by extended periods of mild work softening. The tensile stress-strain behavior of Al-7.5Mg with 15% coarse-grain exhibited somewhat improved plastic deformation and work softening. The ductility and toughness increased over 200% with coarse-grain content of Al-7.5Mg alloys while the yield stress just decreased about 15% drop. The peak flow stresses of the Al-7.5Mg with 0%, 15% and 30% coarse-grain were about 850 MPa, 760 MPa and 700 MPa at room temperature, respectively, which were remarkable for an aluminum alloy. Elongation to failure of Al-7.5Mg with 30% coarse-grain was about 7 % at room temperature. However, unlike pure ultrafine-grained materials, the strength of the present material cannot be attributed solely to grain size refinement. The small degree of work hardening and work softening is not well understood, but may be related to a change in deformation mechanism stemming from the unusual structure.

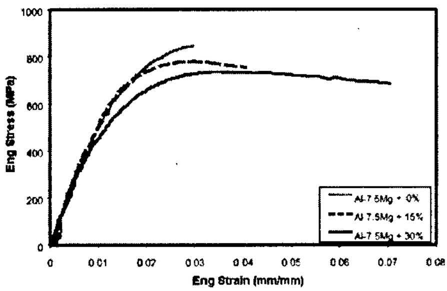

Figure 3. Uniaxial tensile stress-strain behaviours of as-extuded Al-7.5Mg alloy with 0%, 15% and 30% coarse-grain content at room temperature. [4]

The Vickers hardness of bimodal Al-7.5Mg along the extrusion direction was decreased with increasing coarse-grain content from 0% to 30% in figure 4 (b). The hardness is inversely proportional to the coarse-grain content. The ductile coarse-grain phase contributed to decrease the hardness of bimodal Al-7.5Mg using heavy 1 kgf load. The micrograph of 1 kgf indentation on Al-7.5Mg with 30% coarse-grain, as shown in figure 4 (a), is an anomaly, which is unlikely to indent on uniform structure materials. The ductile coarse-grain region showed barrel-shaped indentation faces, which resulted from piling up of the coarse-grain around the faces of indenter, in noticeably contrast to the perfect indentation of nanocrystalline region around the faces of indenter. The hardness of individual coarse-grain and nanocrystalline regions was estimated using a very light 10 gf load, which enabled to indent on coarse-grain region and nanocrystalline region separately as shown in figure 5. The hardness of nanocrystalline region was same on Al-7.5Mg with 15% and 30% coarse-grain, while the hardness of coarse-grain region was decreased and the hardness difference between nanocrystalline and coarse-grain region was increased with increasing coarse-grain content. The hardness of nanocrystalline region was not sensitively related to coarse-grain content. That claims the effective indentation zone of nanocrystalline region was restricted in relatively short range and did not reach to coarse-grain region. Thus, the nanocrystalline region is likely to dissipate its deformation energy in short range. However, the hardness of coarse-grain region was directly depending on the coarse-grain content. The

deformation of ductile coarse-grain region was expanded to nanocrystalline region extensively, which might constraint the deformation of coarse-grain region. The feature is evident on figure 5. The size of plastic zone beneath a hardness indentation will be estimated in ongoing research. The hardness of Al-7.5Mg with 0% coarse-grain was lower than that of nanocrystalline region on Al-7.5Mg with 15% and 30% coarse-grain because Al-7.5Mg with 0% coarse-grain contained a few percent of residual coarse-grain during processing. It might affect small indentations. Consequently the hardness of bimodal microstructures comprised of nanocrystalline grains and coarse-grain bands was depending on coarse-grain content. Moreover, these structures played a significant role in enhancing ductility and toughness by its unusual deformation mechanism in the bulk nanocrystalline alloys.

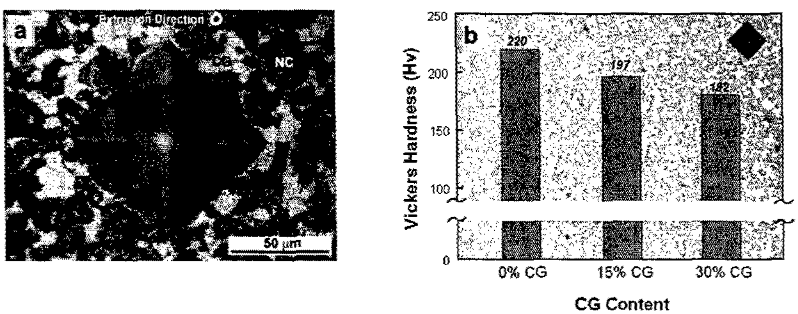

Figure 4. (a) The micrograph of indentation using 1 kgf load on Al-7.5Mg alloy with 30% coarse-grain content along the extrusion direction, (b) Vickers hardness results using 1 kgf load on Al-7.5Mg alloy with 0%, 15% and 30% coarse-grain content along the extrusion direction.

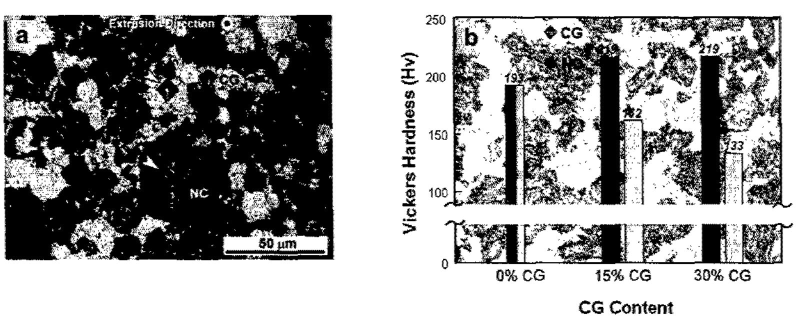

Figure 5. (a) The micrograph of separate indentations using 10 gf load on coarse-grain and nanocrystalline region of Al-7.5Mg alloy with 30% coarse-grain content along the extrusion direction, (b) Vickers hardness results using 10 gf load on coarse-grain (CG) and nanocrystalline (NC) region of Al-7.5Mg alloy with 0%, 15% and 30% coarse-grain content along the extrusion direction.

CONCLUSIONS

In an effort to enhance ductility and toughness of bulk nanocrystalline metals, bimodal structures of Al-7.5Mg comprised of nanocrystalline grains and elongated coarse grains were produced by consolidation of cryomilled powders successfully. Coarse grains were elongated in the extrusion direction and formed ductile coarse-grain bands. Elastic-nearly perfectly plastic stress-strain behavior was observed in tension tests of bulk nanocrystalline Al-7.5Mg with 30% coarse-grain. The unusual absence of work hardening was a noteworthy characteristic of the bimodal Al-7.5Mg alloy. Although the phenomenon is not fully understood at present, it has been reported in other nanocrystalline alloy systems prepared by mechanical alloying, and warrants further investigation. The hardness properties of bimodal Al-7.5Mg alloy revealed the different hardness and deformation behavior between nanocrystalline region and coarse-grain region. Investigation of tensile and hardness test proposes unusual deformation mechanisms and interactions between ductile coarse-grain bands and nanocrystalline regions. The present work has shown that the yield strength can be substantially increased while retaining reasonable ductility, although it was by no means fully optimized. The bimodal microstructure and its ductility toughening effect inspire us to design microstructures and to guide the selection of processing parameters leading to optimal performance characteristics on various nanocrystalline metals.

ACKNOWLEDGEMENTS

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